Thompson River Survey. Chemical Analysis of Water, Tissue and Sediment Samples Collected August 1981 to March 1982

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ABSTRACT

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The three major point-source wastewater discharges to the Thompson River are the Weyerhaeuser Canada Ltd. bleached kraft mill, the City of Kamloops municipal sewage treatment plant and the Gulf Oil refinery. Grab samples of the first two sources were collected for chemical analysis in September and November 1981 and in March 1982. A single sample of oil refinery effluent was collected in May 1982. Populations of rainbow trout and mountain whitefish were sampled in the Thompson River below Kamloops Lake and upstream of McLure on the North Thompson River and near Chase on the South Thompson in August 1981. The fish were sampled a second time in March 1982 during low water flow conditions. Chlorinated compounds were found only in the pulp mill wastewater, which contained several chlorinated phenols, guaiacols and catechols, chlorinated benzoic and dehydroabietic acids and a number of unknowns. Hydrocarbons and monohydric phenols were observed in municipal and oil refinery effluents, which are treated together in an aerated lagoon. Bile and liver tissue extracts prepared from the fish did not contain chlorinated organics or compounds that appeared to originate from the pulp mill. However there was evidence of sewage pollution in both the main stem Thompson and the North Thompson systems. The presence of a series of petroleum hydrocarbons in the Thompson River and South Thompson River fish and a river water sample may be related to public complaints about tainted fish. A sample of sediment from Kamloops Lake, which is downstream from the pulp mill, did not contain identifiable chlorinated organic compounds.

RÉSUMÉ

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Les trois principales sources d'eaux usées déversées dans la rivière Thompson sont l'usine de pâte blanchie au sulphate de la Weyerhaeuser Canada Ltd., l'usine de traitement des eaux usées de Kamloops et la raffinerie Gulf Oil. Des échantillons provenant des deux premières sources ont été recueillis à l'aide d'une benne preneuse en septembre et novembre 1981 et en mai 1982, pour fins d'analyse chimique. Un seul échantillon de l'effluent de la raffinerie de pétrole a été prélevé en mai 1982. Les populations de truite arc-en-ciel et de ménomini de montagnes de la rivière Thompson ont d'abord été échantillonnées en août 1981 en aval du lac Kamloops et en amont de McLure, dans la Thompson-nord, et près de Chase, dans la Thompson-sud, et une deuxième fois en mars 1982 quant le débit était peu élevé. Seules les eaux usées déversées par l'usine de pâte contenaient des composés chlorés soit plusieurs phénols chlorés, guaicols et catéchols, des acides benzoïques et déhydroabiétiques chlorés et un certain nombre d'éléments inconnus. Des hydrocarbures et des phénols monohydriques étaient présents dans les effluents de la raffinerie de pétrole et dans les eaux d'égout qui sont traités ensemble dans une lagune aérée. Les extraits de foie et de bile de poissons ne contenaient pas de composés organiques chlorés ni de composés qui semblent provenir de l'usine de pâte. Il y avait toutefois des signes de pollution due aux eaux usées dans la partie principale de la rivière Thompson et dans la rivière Thompson-nord. La présence d'une variété d'hydrocarbures de pétrole dans les poissons capturés dans la Thompson et la Thompson-sud et dans un échantillon d'eau de rivière peut être relié aux plaintes logées par des individus au sujet de poissons contaminés. Un échantillon de sédiments recueilli dans le lac Kamloops ne contenait pas de composés organiques chlorés identifiables.

INTRODUCTION

In 1971 when a major expansion of the Weyerhaeuser Canada Ltd. pulp mill at Kamloops was being brought into production, complaints from members of the public regarding foam, colour, excessive algal growth and tainted game fish in the Thompson River and Kamloops Lake (Figure 1) resulted in the organization of a federal/provincial task force to study these environmental problems. This work was completed in 1976 with the publication of a large report (Anon 1976) which presented a mass of scientific data on the Thompson River system but did not suceed in defining cause and effect relationships. Although foam problems have not recurred, colour in the receiving waters and nuisance growths of benthic algae have continued as aesthetic problems and contentious public issues. A later, more detailed study by NWRI (Environment Canada) in cooperation with Weyerhaeuser Canada Ltd has clearly established the role of phosphorus discharged from the pulp mill and the City sewage lagoons as the agent responsible for downstream algal blooms (Bothwell and Daley 1981).

To assess the current environmental health of the Thompson River, below Kamloops Lake, Environmental Protection Service did a field study in the area in August 1981. Water and sediment samples were collected from stations on the Thompson River and on Kamloops Lake, respectively. Resident rainbow trout (Salmo gairdneri) and mountain whitefish (Prosopium williamsoni) were taken by beach seine, gillnet or angling from the North Thompson River upstream of McLure, the South Thompson near Chase and from the Thompson River near Walhachin (Fig. 1) in August 1981. Fish were again sampled at Walhachin by the British Columbia Fish and Wildlife Branch in March 1982 during low water flow.

This report describes the results of analyses performed in this laboratory in support of the study with emphasis on a search for bioaccumulation of chlorinated organics in freshwater fish species from the Thompson River system.

EXPERIMENTAL METHODS

Sample extract preparation

Fish

Fish muscle tissue samples were received from the field in frozen condition and wrapped in heat-treated tinfoil. The livers and gallbladders were separately packaged in heat-treated glass sample jars.

Tissue samples were disintegrated in water using a Brinkman polytron. The homogenate was acetylated according to the procedure described by Voss et al (1981). Typically a homogenate of 7.5 g liver tissue was reacted with acetic anhydride (10 mL) in the presence of potassium carbonate (4 g). After stirring at room temperature 1 hr, the mixture was extracted with hexane (3x25 mL). A

blank was prepared using all the above ingredients except the tissue. The hexane extracts were reduced to 1 mL volume in a Kuderna Danish apparatus.

The spent reaction mixture from the acetylation was acidified with sulfuric acid, extracted with ether (3x25 mL), the extract concentrated as before and methylated with diazomethane.

Sediment

Sediment samples from six stations in Kamloops Lake were combined. The wet sediment (284 g) was acetylated as described above and the reaction mixture extracted three times with hexane (3x75 mL). The combined extracts were reduced as before to 1 mL volume. The sample was split into two equal portions and one was methylated with diazomethane.

Wastewater and River Water

Water samples were shipped in 10-gallon glass containers which had been rinsed with water, acetone and hexane. Typically a sample of treated mill effluent (1 L) was adjusted to pH 10 and extracted with methylene chloride (100, 75 and 50 mL). The extracts were combined and reduced to 1 mL.

A second sample of mill effluent (1 L) was acetylated by the procedure already described.

A river water sample (2 L), collected on March 24, 1982 at Ashcroft, 37 km below the lake, was extracted with methylene chloride at pH 10 to yield a base/neutral extract and again at pH 2 to give an acid extract which was methylated with diazomethane.

Base/neutral and methylated acid fractions were also prepared from Kamloops municipal sewage samples using both methylene chloride and ether as extracting solvents in separate trials. A sample of wastewater from the Gulf Oil refinery was processed similarly except that base/neutrals were extracted first with methylene chloride and then with ether. The acid fraction was extracted only with ether.

Analysis by Gas Chromatography - Mass Spectrometry (GC-MS)

All extracts were analysed using a Hewlett-Packard Model 5992 B GC-MS instrument fitted with a capillary column injection system and cryogenic cooling device. The oven temperature was programmed from 30° to 290°C at 10°/min. The ion source was maintained at 70 electron volts and the instrument was operated in the peakfinder mode using perfluorotributylamine for automatic internal calibration. The various fused silica capillary columns were: a) 30 m x 0.32 mm DB 5 bonded phase (J & W Scientific); b) 25 m x 0.30 mm OV 101 (Hewlett Packard); and c) 25 m x 0.30 mm SE 54 (Hewlett Packard). Mass spectra were identified by manual searching of the Eight Peak Index Aldermaston File and the EPA - NIH File. Some compounds were confirmed by comparing the mass spectra and retention times of standards with those observed in the extracts.

RESULTS AND DISCUSSION

Pulp mill wastewater analyses

The analyses of treated wastewater samples from the Weyerhaeuser Canada Ltd. bleached kraft mill are summarized in Table 1. Grab samples were collected in September and November 1981, and in March 1982. On all three occasions numerous chlorinated organics were detected, including compounds of the phenolic and carboxylic acid classes and the neutral substance dichloromethyl methyl sulfone. In every case several chlorinated organic compounds, for which no published mass spectra are currently available, were also detected. Given their low concentrations in the wastewater, isolation and identification using other procedures would entail difficulty and effort beyond the scope of the present work.

Many nonchlorinated organics were also present in the wastewaters but have not been included in Table 1 because the objective of the study has been to concentrate on chlorinated compounds which may have a tendency to bioaccumulate in the foodchain. These other nonchlorinated compounds belong to the chemical classes of the fatty acids, resin acids, mono- and diterpenes. Among the terpenes we observed the presence of hydrocarbons, alcohols and aldehydes.

We are presently unable to quantify the chlorinated substances present in a bleached kraft wastewater sample because the necessary standards are unavailable. Consequently, these standard compounds would have to be synthesized or acquired from other researchers and as well, their retention times, response factors, detection limits and recovery values from wastewater established. However the biological oxidation system at Weyerhaeuser Canada appears to work efficiently as concentrations of individual resin acids in treated wastewater were below 1 ppm. On that basis most of the chlorinated organics detected are in the 10 to 100 ppb concentration range in the effluent.

The presence of chlorinated catechols and guaiacols is not surprising as these have been observed in the wastes of many bleached kraft mills and are derived from the residual lignin still attached to the pulp fibres following the digestion process. That many of these chlorinated phenolic substances survive the biological oxidation treatment is also in accordance with what has been observed elsewhere (Voss et al. 1981; Leach 1979). Escapement of such compounds to the receiving waters raises the possibility of biomagnification in the foodchain. Such compounds have been observed to bioaccumulate in fish exposed under laboratory conditions (Landner et al. 1977; Renberg et al. 1980). However it is not clear how seriously this affects the survival and performance of fish in the receiving environment. Depuration rates have been shown to be of the order of ten days when contaminated fish are transferred to clean water (Landner et al. 1977).

Kamloops municipal sewage and gulf oil refinery wastewater

The results of analyses of grab samples of Kamloops municipal sewage, collected in September and November 1981, are summarized in Table 2 together with the data recorded on a single sample of oil refinery wastewater collected in May 1982. The Gulf Oil refinery effluent is treated in the municipal sewage treatment system, which in summer and fall is allowed to discharge to the

Thompson River during high water flow conditions. These discharges to the river ceased on November 30, 1981 and commenced again on March 4, 1982. During this period of the year the river water traverses Kamloops Lake as a surface overflow because of density differences (Anon 1976) and this cold-season-phenomenon is used as a strategy to protect the Thompson River below the lake from environmental effects caused by high nutrient concentrations in the ambient water.

No chlorinated organics were detected in sewage or oil refinery wastewaters. City sewage samples contained a series of common fatty acids, several aromatic carboxylic acids including two isomers of phthalic acid, the basic compounds nicotine and caffeine and a basic compound presumptively identified as indole, plus several neutral substances such as α-terpineol, cyclohex-2-enone, naphthalene and the fecal sterols coprostanol and cholesterol. The compound α-terpineol is widely used in household cleansing formulations and we have found it to be the most abundant of the detectable organics typical of municipal wastewaters.

Two other samples of sewage contained members of two homologous series of saturated paraffinic and olefinic hydrocarbons and some antioxidants plus the heterocyclic compounds benzothiazole and dimethylacridan. The structures of several of these latter compounds suggests that they originate in the oil refinery. Furthermore they are not mentioned in published work on the composition of raw and treated municipal wastewater (Garrison et al. 1976).

A single grab sample of oil refinery wastewater was collected from Gulf Oil in Kamloops on May 5, 1982. This sample contained no detectable chlorinated organics and its most notable feature was the presence of a series of at least nineteen paraffinic and olefinic hydrocarbons. Also detectable were three low-molecular-weight alkylphenols, a monomethylnaphthalene and two dimethylnaphthalenes. An unusual compound present was N-acetylmorpholine which must have some specialized use in the refinery operation - perhaps in leak detection. Apart from two fatty acids and dimethyl sulfone - a byproduct or metabolite from heterocyclic sulfur compounds present in crude oil - no other organics were detected in this wastewater.

Fish liver and bile samples

The analyses of liver and bile samples of fish collected from the Thompson and the North and South Thompson are summarized in Table 3. Only rainbow trout were available from the South Thompson. No chlorinated organic compounds were detected in a sample (14.5 wet weight) of pooled liver tissue from four adult rainbow trout from the Thompson, nor from corresponding samples of bile, skin or muscle. Apparently fish exposed to chlorinated catechols and guaiacols still contain at least some unconjugated phenolic material (Ladner et al. 1977) so that saponification of the extracts is not essential for detection of such compounds. Extracts of pooled liver and bile tissue samples from four mountain whitefish also failed to reveal the presence of chlorinated organic compounds.

A composite sediment sample, prepared from six cores taken in Kamloops Lake, was acetylated and analyzed. The extract was also negative for the presence of chlorinated phenolic compounds. A diterpene hydrocarbon - sandaracopimaradiene - was observed. This is a wood extractive which may have entered the receiving environment by either natural phenomena or via the

activities of the forest industry. The compound was not present in a blank run on the acetylation procedure. Compounds of the hydrocarbon class are more hydrophobic than are phenolics and hence have higher octanol/water partition coefficients. It is thus not surprising to find a diterpene hydrocarbon concentrated in the sediment, whereas the chlorinated guaiacols and catechols are more likely to remain predominantly in the water column.

During the period July 14 to August 30, 1981 the Weyerhaeuser Canada Ltd. pulp mill was closed due to a strike. This coincided with the field sampling program conducted by Environmental Protection Service. At this time of the year the calculated turnover time for the water in Kamloops Lake is about 28 days (Anon 1976). It has been observed that fish contaminated with chlorinated guaiacols and catechols cleansed themselves in about 10 days when transferred to clean water (Landner et al. 1977). There is thus a possibility that the fish might have purged themselves of chlorinated contaminants during the strike. Accordingly the fish were resampled in the Thompson and North Thompson Rivers during March 1982 at low water.

Bile samples from two whitefish taken in March 1982 from the North Thompson were pooled, analyzed and compared to similar samples from five fish collected in the Thompson. The gas chromatographic profiles of these extracts were extremely complex and mass spectra of many components were impossible to interpret because they did not reveal the parent ions. Most of these compounds are probably from the fishes normal metabolic pool and should be removable by suitable extract cleanup procedures. It is difficult to be certain which components of the extracts are pollutants given the lack of literature data on normally occuring compounds in mountain whitefish and because of the unavailability of an unpolluted control sample. In this regard it would have been beneficial to have had samples of whitefish collected from the South Thompson River system for analysis. Liver extracts from whitefish taken at both locations were also analyzed and were less complex in composition but contained larger proportions of fatty acids.

Again all bile and liver extracts gave negative results for the presence of chlorinated organics. However, two other types of pollution may be affecting the fish that were analyzed. The first was sewage pollution, which seemed to affect both the main stem Thompson and North Thompson fish but was more prevalent in the former. This influence was indicated by the presence of benzoic acid, phenylacetic acid, cyclohex-2-enone, nicotine, anthranilic acid and phthalate esters. These compounds have been reported by Garrison et al (1976) in municipal sewage and by ourselves in current unpublished studies at Iona and Annacis Island Sewage Treatment Plants which service Greater Vancouver. They were also seen in Kamloops municipal sewage (Table 2). The North Thompson fish sampling stations were upstream of McLure but other small communities, farther upstream, also discharge to the North Thompson. It is not known whether agricultural runoff from cattle ranching in the river valley may also contribute to this pollution.

The other potential pollution problem noted was the presence of a series of at least 22 paraffinic hydrocarbons in liver tissue samples from the main stem Thompson River. This was clearly parallelled by the presence of a similar series of compounds in a water sample collected on March 24 1982 at

Ashcroft below Kamloops Lake. Although the oil refinery may contribute paraffinic hydrocarbons to the Thompson, it is not the only source as many small industrial operations exist in the Thompson Valley. Furthermore similar compounds were identified in rainbow trout livers from the South Thompson, which is not close to industrial operations.

Other compounds listed in Table 3 seem to be of miscellaneous origin. Xylene and cumene are used as solvents and as components of fuels. Cyanuric acid is used as a selective herbicide and in swimming pool disinfection. Methyl carbitol is a water-soluble solvent. No uses for cyclohexyl benzene or 1,2-dihydro-1,5,8-trimethylnaphthalene are known.

It had been proposed that fish be exposed under controlled laboratory conditions to dilutions of Weyerhaeuser Canada bleached kraft treated wastewater. This would have allowed demonstration of the bioaccumulation of chlorinated organic compounds under worst conditions and to study their rates of depuration. This experiment was not included in the study and it would appear that further work of the present type should not be performed without evaluating the biomagnification potential of the chlorinated organics in algae, fish and invertebrates as well.

Although the present work is neither exhaustive nor conclusive, it does suggest that some chemical pollution of the freshwater fishery in the Thompson and North Thompson Rivers is occurring but the degree of pollution cannot be estimated from the present findings. This may indicate the need for a more thorough evaluation of the inputs to the system, particularly in relation to the problem of tainting in the sports fishery of Kamloops Lake and the main stem Thompson River.

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Table 1. Chlorinated Compounds Identified in Weyerhaeuser Canada Treated Wastewater.

Sample date	Chlorinated compounds identified	Chlorinated compounds not identified	Chromatographic column
September 29 1981	Dichlorophenol Trichlorophenol Tetrachlorophenol Dichlorocatechol Trichlorocatechol Tetrachloroguaiacol Tetrachlorocatechol Dichloroacrylic acid Dichloromethyl methyl sulfone Dichloromaleic acid (2 isomers) Trichloroacrylic acid 4-Chlorobenzoic acid 2,4-Dichlorobenzoic acid Monochlorodehydroabietic acid Dichlorodehydroabietic acid	10	b
November 6 1981	Dichloromethyl methyl sulfone 4-Chlorobenzoic acid Dichlorophenol Trichlorophenol 2,4-Dichlorobenzoic acid Trichloroguaiacol (2 isomers) Tetrachloroguaiacol Dichlorocatechol (2 isomers) Trichlorocatechol Tetrachlorocatechol Dichloroacrylic acid	13	b
March 24 1982	Dichlorophenol Trichlorophenol Trichloroguaiacol (2 isomers) Tetrachloroguaiacol Dichlorocatechol Trichlorocatechol Dichloromethyl methyl sulfone Chlorobenzoic acid (2 isomers) 2,4-Dichlorobenzoic acid Chlorodehydroabietic acid (2 isomers) Dichlorodehydoabietic acid	6	a

Table 2. Compounds identified* in Kamloops City Sewage Treatment Plant and Gulf Oil Refinery wastewaters.

Sampling Date	Type	Compounds identified	Unknown	Chromatographic column used
September 29 1981	Kamloops City Sewage	1,5-ditertiarybutyl-3,3-dimethyl-bicyclo (3.1.0) hexane ditertiary butylhydroxytoluene dimethylacridan dehydroabietic acid 2 phthalate esters coprostanol cholesterol series of 19 paraffin hydrocarbons	10	c
November 6 1981	Kamloops City Sewage	benzoic acid 11 fatty acids benzothiazole 2 phthalate esters 6 paraffin hydrocarbons	16	c
March 24 1982	Kamloops City Sewage	Cyclohex-2-enone 2-ethylhexanoic acid benzoic acid glutaric acid dimethylphenol phenylacetic acid α-terpineol hydroxybenzoic acid indole phenoxyacetic acid anthranilic acid nicotine 2-hydroxy-3 methylbenzoic hydroxybenzoic isomer phthalate ester naphthalene 2 phthalic acid isomers d_{10} anthracene (internal std.) caffeine 6 fatty acids	68	a

Table 2. (cont)

Sampling Date	Type	Compounds identified	Unknown	Chromatographic column used
May 4 1982	Gulf Oil Refinery	dimethyl sulphone difluorobenzoic acid (internal std.) m-cresol o- and p- cresol dibromophenol (internal std.) lauric acid palmitic acid ethylphenol N-acetylmorpholine monomethylnaphthalene 2 dimethylnaphthalenes dibromodimethoxybenzene (internal std.) series of 19 paraffinic hydrocarbons	1	a

* No chlorinated compounds identified.

Table 3. Analysis of rainbow trout and mountain whitefish tissues.

Pollutants observed	Tissue*	Date	Location and sample size ( ) Thompson	North Thompson	South Thompson
Organochlorines	W, B&L	Aug. 1981	Absent (4)	not analysed	--
Organochlorines	R, B,L, S&M	Aug. 1981	Absent (4)	not analysed	Absent (2)
Xylene	W, B&L	Mar. 1982	+ (5)	+ (2)	
Methyl carbitol	W, B&L	" "	+	+ #	
Cyclohex-2-enone	W, B	" "	+	-	
Benzoic acid	W, B&L	" "	+	weak #	
Cyclohexylbenzene ?	W, B	" "	+	-	
Nicotine	W, B	" "	+	-	
Cyuanuric acid	W, B	" "	-	+	
2 phthalate esters	W, B&L	" "	+	-	
Paraffin hydrocarbons	W, B&L	" "	+	-	
2-methylcyclobutanone ?	W, L	" "	+	-	
Cumene	W, L	" "	-	+	
1,2-dihydro-1,5,8-trimethylnaphthalene ?	W, L	" "	-	+	
Phenylacetic acid	W, L	" "	-	+	
Anthranilic acid	W, L	" "	-	+	
Paraffin hydrocarbons	R, L	Sept. 1981			+

* R = rainbow trout; W = mountain whitefish
B = bile; L = liver; S = skin; M = muscle

North Thompson samples contained these compounds only in liver tissues.

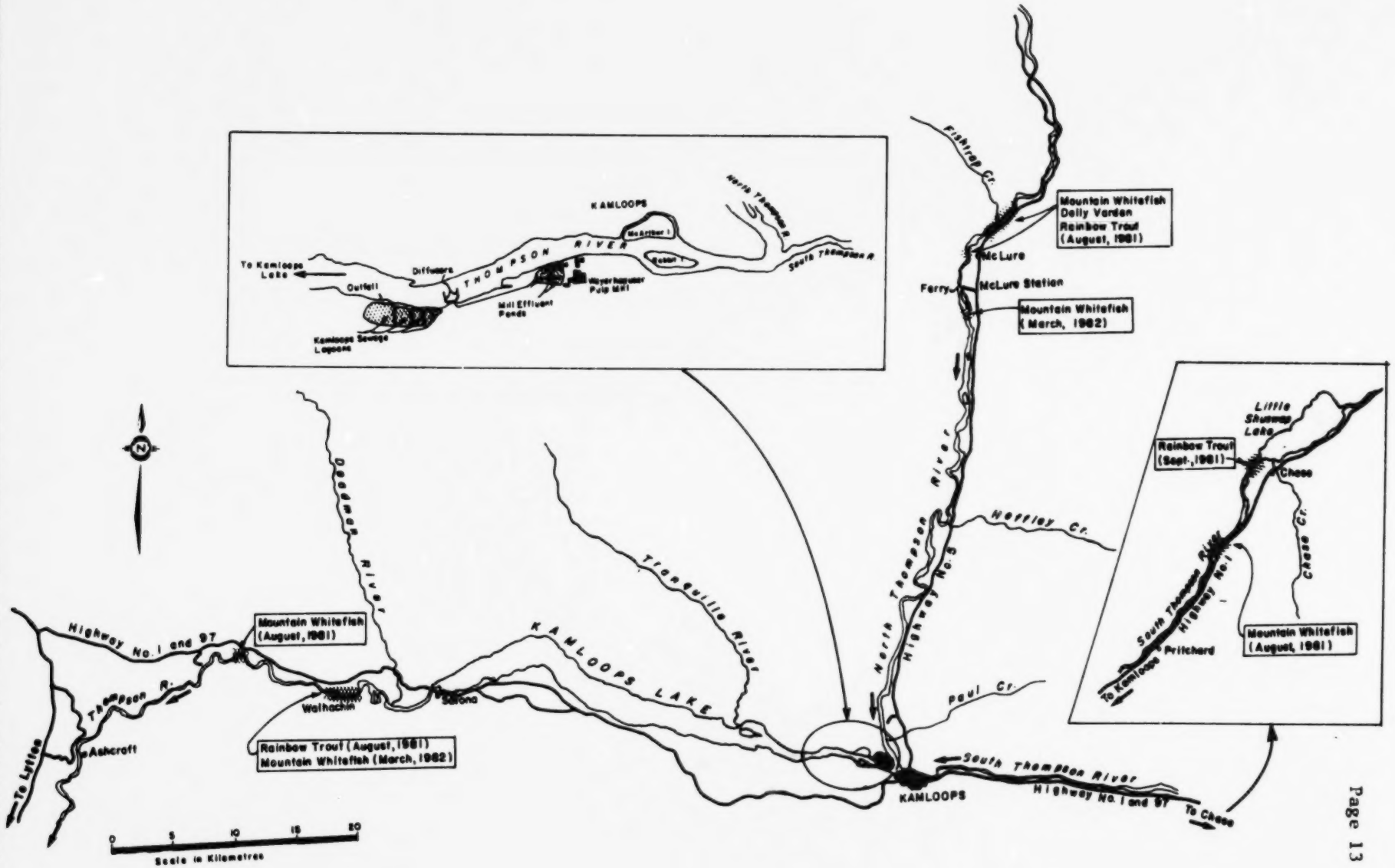

FIGURE 1 GENERAL LOCATION OF AREAS WHERE FISH WERE COLLECTED